A CLAY MINERALOGICAL STUDY OF CERTAIN REDDISH PRAIRIE SOILS OF OKLAHOMA WITH AN ESTIMATION OF THE MONTMORILLONITE AND ILLITE CONTENT

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF ILLUSTRATIONS

INTRODUCTION

The clay fraction of soils has long been recognized as playing an important part in determining the properties of soils.

The permeability of soils to air and water, their adaptability to specific crops, their fertility (except nitrogen), their absorptive power, and their tilth are all influenced to a marked degree by the colloidal constituents.

A knowledge of clay mineralogy aids in the study of soil weathering processes and soil profile development, aids in characterizing a soil type and provides an additional tool in the correlation of soil types, and aids in the classification of those areas where problems of soil physical condition are particularly acute.

The identification of clay minerals in the soils of Oklahoma has not, as yet, been fully investigated. A knowledge of the clay mineralogy in problem soils, especially the soils with dense clay subsoils such as Kirkland, would be an additional tool for use in soils research.

Soil classification in this State would be aided. Clay mineralogy helps in distinguishing the Great Soil Groups and aids in relating a soil to its parent material.

PURPOSE OF STUDY

The quantitative estimation of the clay minerals would be an aid in the study and evaluation of research and classification of Oklahoma soils.

The specific purpose of this study was to estimate the percentages of Montmorillonite and Illite in the clay fraction of the B and C horizon of three selected soil sites. The sampling sites were selected in a north-south direction to observe any effect of location on the mineralogical composition of the soils.

A secondary purpose of the investigation was to study the effect of the separation of the clay-size material into three fractions with the supercentrifuge.

REVIEW OF LITERATURE

The Clay Minerals

According to Grim, (15), mineralogical analysis of clays show that most clays fall into three main series - Kaolinite, Montmorillonite, and Illite or Hydrous Mica.

Kaolinite: The most common member of this series is Kaolinite proper with the composition of $Al_4Si_4O_{10}(OH)_8$ (15,17). Other members of the Kaolinite series are Dickite and Nacrite with similar compositions but of different crystallographic forms. Another member, Anausite, differs from Kaolinite by having a higher silicon and a lower aluminum content.

Ross and Kerr (31) have shown that Halloysite is closely related to Kaolinite but it has a distinct x-ray pattern and dehydration curve.

The structural unit of Kaolinite appears to be a double sheet composed of a gibbsite layer (aluminum in octahedral coordination) and a Si-O layer (silica in tetrahedral coordination) (17).

Montmorillonite: The minerals in this series include: Montmorillonite proper, Beidellite, Nontronite, and Saponite. Montmorillonite proper has the composition, $Al_2Si_4O_{10}(OH)_2.xH_2O$ with magnesium usually present (17). Beidellite has a lower silicon and a higher aluminum content which might suggest a small amount of replacement of Si^{+4} by Al^{+3} in the silica tetrahedral sheets. Nontronite has some of the Al^{+3} of the gibbsite layer replaced by Fe^{+3}. Saponite has all of the aluminum replaced by magnesium.

Montmorillonite consists of structural units of one gibbsite sheet between two sheets of tetrahedral silica groups (15,27). The structural

units are loosely held together with H_2O present between them.

Illite: The structure of Illite is intermediate between Montmorillonite and Muscovite (17). It is similar to Montmorillonite in that it also consists of units of one gibbsite sheet between two sheets of tetrahedral silica groups (15). Potassium is the interlayer ion which forms a strong bond between the Si-O layers. It tends to prevent adsorption of water and limit the base-exchange capacity of the interlayer ions (27). The composition of Illite as given by Hendricks and Alexander (17) is $KAl_2AlSi_3O_{10}(OH)_2.xH_2O$.

Identification of Clay Minerals

Some of the methods used in identifying clay minerals include: X-ray analysis, dehydration curves, differential thermal analysis, optical, electron microscope, chemical analysis and cation-exchange capacity determinations.

The optical method is one of the oldest means of studying clay minerals (14). It involves the use of refractive indices which are not especially well adapted to soil colloids. The indices are an average for a mixture of minerals which are usually present in the soil.

X-ray analysis is considered the most accurate means of identification (24). Some difficulty is involved in differentiating Illite or Hydrous Mica diffraction patterns from those of Montmorillonite (18).

Dehydration involves the loss of chemically held water in the clay lattice upon heating (24). This test is not accurate for quantitative estimation of clay minerals.

Differential thermal analysis is the study of the exothermic and endothermic reaction of clays when they are being gradually heated (27).

Each clay mineral has its characteristic thermal curve. Quantitative determination can be made by comparing the intensity of the thermal "pips" with known standards of mixtures of the clay minerals.

Methods Used in This Investigation

Ethylene glycol retention: Dyal and Hendricks (11) introduced a simple gravimetric method for measuring total surface, external surface, and, by difference, internal surface of clays based on their retention of ethylene glycol. The only clay minerals that form definite solvates with polar molecules between the structural layers are Montmorillonite, Vermiculite and Endellite (4,11). The latter two minerals are not too important as constituents of the soil colloid.

One molecule of ethylene glycol displaces two molecules of water and forms a two layer solvate, in the presence of excess solvent, between the lattice units in Montmorillonite (5,28). Ethylene glycol does not displace exchange ions, but does assume a single layer configuration on the plane of the clay surface. The glycol is held to the clay by an attraction in the nature of a C-H···O bond between methylene groups and the oxygen surface of the clay (5).

Dyal and Hendricks (11) added an excess amount of ethylene glycol to a dried sample of clay and gravimetrically determined the retained glycol after distillation of the glycol-clay under vacuum for periods of sixteen to twenty-four hours. The distillation left a two-layer glycol solvate between the lattice units and a one layer solvate on the external surface of the clay particles. Another clay sample was heated to 600°C for four hours. This treatment resulted in the destruction of the interlayer swelling. Under distillation with glycol, these clay particles retained

only a one layer glycol configuration on the external surface. Therefore, total glycol retention (total surface) minus external glycol retention (external surface) equals internal glycol retention, hence the amount of interlayer swelling. The results can be normalized against a known sample of Montmorillonite, and the values obtained used as an index of the percentage of Montmorillonite type clay in the colloidal fraction of the sample.

Mica-like clay minerals may have interlayer swelling to the extent of about ten per cent of that of Montmorillonite. Kaolinite has very low glycol retention due to a small surface area and no interlayer swelling.

Some difficulties are involved in the ethylene glycol retention method. Ethylene glycol may form from one to two layers on the external surface of the clay particle due to the close proximity of other clay particles. Dyal and Hendricks (11) show that more glycol is retained than that which is calculated for a one layer configuration. The error would be small with Montmorillonite due to a relatively small external area (only about 5% of the total surface area) in this clay mineral.

Another uncertainty is the loss of glycol from the clay particle under vacuum distillation. This loss is continuous but at a low rate after the excess glycol has evaporated. Twenty-four hours of distillation was used by Dyal and Hendricks (11) with soil samples.

Destruction of interlayer swelling by heating has inherent dangers. Kaolinite is decomposed but the total surface area is not greatly changed. Hydrogen saturated Montmorillonite, after being heated to 600°C, has a surface area in the same order of magnitude as the values found for Kaolinite.

The lack of separation of some layers in Montmorillonite would result

in a lower glycol retention. Dyal and Hendricks (12) show that certain cations, such as potassium, can reduce glycol retention in Montmorillonite by restriction of the interlayer swelling.

Bower and Gschwend (4) modified the ethylene glycol retention method of Dyal and Hendricks to apply more specifically to soils. The greatest differences between the two methods are the pretreatment of the sample and the time of heating to destroy interlayer swelling. Bower and Gschwend used the whole soil, without any separation of the clay fraction, for the sample, whereas Dyal and Hendricks separated the clay fraction from the sand and silt. Bower and Gschwend heated their sample for two hours to destroy interlayer swelling while Dyal and Hendricks had a heating pericd of four hours. Other refinements were made in order to made the procedure more suitable for use on soil samples. The analytical portion of this investigation was completed before Bower and Gschwend published their procedure.

Bower and Gschwend studied the effects of various exchangeable cations, organic matter, and soluble salts upon ethylene glycol retention. They found that a four hour period of heating at 600°C may increase the glycol retention of soils that contain appreciable amounts of alkaline earth carbonates. Their data show that a heating period of two hours permitted a high degree of interlayer suppression without significant interference from these carbonates.

Bower and Gschwend found that the normal cation composition on the exchange complex on most soils would not greatly influence the total glycol retention. Saturation of the exchange complex with either potassium or ammonium, however, would depress glycol retention.

They found that organic matter also has glycol retention properties and must be removed from the sample to eliminate interference.

Soluble salts were found to have little effect upon glycol retention when calculations were made on the basis of salt free soil.

Base Exchange Capacity: Clay minerals possess the ability to adsorb cations and under proper conditions these cations are exchangeable for other cations.

Montmorillonite has a high base exchange capacity due to replacement of ions within the lattice. Mg^{+2} replaces Al^{+3} in the gibbsite layer thereby providing an excess negative charge on the lattice. Replacement of slightly less than 20 per cent of the Al^{+3} positions with Mg^{+2} in the gibbsite layer would provide a base-exchange capacity of about 100 m.e. per hundred grams (15).

Another replacement can occur in Montmorillonite. Si^{+4} can be replaced by Al^{+3} in the silica tetrahedral sheets. This replacement is minor in the Montmorillonite series (15).

Some of the exchange capacity in Montmorillonite can be attributed to broken bonds at the edge of fragments. Shaw (35) reports that the exchange capacity of Montmorillonite can be increased by grinding which increases the number of edges.

Montmorillonite is built up of loosely held units and can expand and readily cleave into extremely thin flakes upon agitation in water; thus, a huge surface area becomes available to hold exchangeable cations. Hauser and Reed (16) found that the base exchange capacity of Montmorillonite does not vary with particle size. Shaw (35) states that Montmorillonite particle size depends on the degree of dispersion. Ultimate dispersion would separate all Montmorillonite particles into plates of unit thickness.

The exchange capacity mechanism in Illite is similar to Montmorill-

onite (27). Illite has more Si^{+4} positions occupied by Al^{+3}. This replacement exists to the extent of about 15 per cent of the Si^{+4} positions. The excess charge resulting from this replacement is satisfied by potassium ions and the units are held together with little power of expansion (15). This lack of expansion between units and subsequent holding together when agitated in water reduces the surface area available for base exchange in Illite.

Replacements generally do not take place in the Kaolinite structure (15). Kaolinite lattice charges are balanced internally; therefore, this mineral is unable to attract cations except by negative charges due to the broken bonds on the edges of fragments (26).

Table 1 presents the base exchange capacities of the various minerals as given by different authors.

Ross and Hendricks (32) state that all the Montmorillonite type minerals have almost the same base-exchange capacity (85 m.e. per 100 grams). Kelly (26) says that soils high in Montmorillonite colloids have rarely been found to exceed 75 m.e./100 grams of colloid. Gieseking (14) states that the inorganic exchange capacity may vary from practically no exchange capacity to 60 m.e./00 grams of soil.

According to Kelly (26), base exchange capacity can only be used as an aid in quantitative estimation of clay minerals. Degree of weathering (19) and error of omission (since most soil colloids usually contain uncombined oxides and hydroxides and colloidal silica) will cause the base exchange capacity to vary.

Total Non-exchangeable Potassium: Considerable disagreement exists among authors as to the percentage of non-exchangeable potassium present in Illite. According to Gieseking (14) most workers assume 6 per cent

Table 1. Base exchange capacity of minerals as reported by authors in m.e. per 100 grams

Author	Minerals					
	Bentonite and Montmorillonite Series					
	Benton-ite	Montmorill-onite	Beidel-lite	Nontron-ite	Attapul-gite	Sapon-ite
Grim (15)		60-100				
Bower & Troug (3)	90		80			
Caldwell & Marshall (8)				57.0-64.3	18.0-22.2	69.3-86.3
Kelly (26)		90-100				
Bradley (5)	80					
American Colloid (2)	89.2					
Kelly and Jenny (23)	126.2					
Jackson, et al (21)		80				

	Illite and Kaolinite Series	
	Illite	Kaolinite
Grim (15)	20-40	3-15
Bower and Troug (3)	24.2	
Kelly (26)	30-40	not more than 10 or 15
Jackson (21)	20	

	Primary and Secondary Minerals	
Kelly and Jenny (23)	Talc	Trace
	Albite	1.0
	Anorthite	2.0
	Biotite	3.0
	Pyrophyllite	4.0
	Orthoclase	5.0
	Chlorite	6.0
	Muscovite	10.0

K_2O. He points out that these assumptions are not strictly valid but that this method appears to be the best available for the estimation of Illite. Alexander, et al (1) say that the K_2O content is a good criterion for estimating the quantity of Hydrous Mica since this is the only mineral which contains potassium. Hendricks and Alexander (17) state that colloids containing over 1 per cent K_2O have Illite as a predominate mineral. Kelly (26) states that K_2O content can give only a rough estimation since the potassium content of Hydrous Mica from different sources has been found to vary considerably. Jackson and Hellman (18) point out that potassium can be held in two forms other than Illite, namely Muscovite and fixation of potassium in Montmorillonite. The amount of K_2O that can be fixed in Montmorillonite in a non-exchangeable form may vary from 0.7 to 0.9 per cent.

Coleman and Jackson (10) and Buehrer, et al (7) assumed 6 per cent K_2O in Illite. Alexander, et al (1), Coleman, et al (9), and Jackson, et al (21) assumed 5 per cent K_2O.

Particle Size of Clay Minerals

According to Coleman and Jackson (10) and Pennington and Jackson (30), Montmorillonite is concentrated in the fine clay, less than 0.08 microns in diameter. Shaw (35) says that Montmorillonite will separate into particles of unit thickness. Wyoming Bentonite (which is 90 per cent Montmorillonite) when dispersed in water, will separate into extremely fine particles with 87-89 per cent finer than 0.5 microns and 60-65 per cent finer than 0.1 microns (2).

According to Pennington and Jackson (30), Illite is found in the 2-0.2 micron range while mica intermediates are found largely in the

0.2-0.08 micron range. Jackson and Hellman (18) state that Illite particles occur extensively in the less than 0.2 micron size fraction and therefore, can not be separated from Montmorillonite by mechanical separation.

Kaclinite is usually concentrated above the 0.08 micron size fraction (30). Coleman and Jackson (10) say that Kaolinite occurs in the particle size group, 0.2-0.08 microns in diameter.

Constituents Other Than Clay Minerals in Soil Colloids

According to Kelly and Page (25) other types of crystalline materials, such as quartz, zircon, and hydrargillite, may be present in the clay fraction. Amorphous materials include: hydrated iron oxides, silica, and precipitated permutite-like substances. Kelly, et al (24) state that quartz is found in all soil colloids. According to Coleman and Jackson (10) quartz is the predominant mineral in coarse clay (2-0.2 microns). Jackson and Hellman (18) say that very little quartz is found in separations smaller than 0.2 microns.

Clay Mineral Weathering

Publications by Jackson, et al (19) and Jackson, et al (21) are excellent references for the weathering sequence of clay-size minerals in soils and sediments. Only a brief general review of Literature will be given here.

Jackson, et al (19) give a sequence of thirteen stages of weathering as represented by type minerals: gypsum, calcite, hornblende, biotite, albite, quartz, illite, mica intermediates, montmorillonite, kaolinite, gibbsite, hematite, and aratase (corundum). The weathering stage of a colloid is considered to be a resultant of intensity factors (temperature,

moisture transfer, acidity, and oxidation-reduction) and capacity factors (particle size and specific nature of the minerals) together with time.

From three to five minerals of the weathering sequence are usually present in the colloid of any one soil horizon. One to three intermediate stages may be absent and one or more stages may occur out of sequence such as secondary depositions, particularly gypsum and calcite. The weathering equations are reversible, moving largely to the right in soils and to the left in sedimentary deposits.

Kelly, et al (24) consider Montmorillonite as being formed in the presence of bases. Ross and Hendricks (32) state that alkaline conditions tend to be Montmorillonitic, and Kerr, et al (27) state that Montmorillonite forms under conditions of low rainfall and poor leaching. These conditions are characteristic of grasslands.

Kelly (26) found that dryland soils often contain much Hydrous Mica-type clay. This is supported by Jackson, et al (21) who found that Illite was a predominate mineral in the following main soil groups of China: desert soil, sierzem, pedical, and noncalcic soil.

Kaolinite forms under nearly the opposite conditions to those under which Montmorillonite is formed. Kaolinite forms from basic igneous rocks under conditions of abundant rainfall and active leaching (27). Coleman and Jackson (10) state that Kaolinite appears to be more predominate in the more mature soil profiles in the Southeastern United States. Kelly, et al (24) say that Kaolinite forms in the absence of bases.

Geology of The Pennsylvanian and Permian Deposits

Sheerar (36) describes the geological formations which are pertinent to this investigation. They are as follows:

Pennsylvanian: Pennsylvanian rocks occupy a broad area in Eastern Oklahoma. In the southern and eastern parts of the area, the formation consists of sandstone and shale. Further north, limestone is interbedded with the shales and sandstones. The Labette shale lies within this area. The outcrops occur in a belt extending south from the Kansas line to Big Creek Valley, then to the junction of Big Creek and Verdigris River, then southwest to Broken Arrow. These shales have been used for bricks.

Shales found in the Pennsylvanian system are quite varible. Some of them swell extensively when wet, while others have little to medium swelling characteristics.

Permian: With few exceptions, rocks of this formation outcrop over the entire western part of the State. The formations are generally known as "Red Beds" because of their color. In general, the "Red Beds" formations consists of red shales and sandstones. The red color of the shales is due to ferric iron which forms a thin coating over the particles. The amount of iron varies from five to twenty per cent. These shales usually have high swelling properties. Considerable gypsum is found in the shales of the higher formations, running as high as fifty to sixty per cent. Dolomite is also found in the Woodward formation.

Buckhannan and Ham (6) state that the Permian "Red Beds" material was reassorted from older sedimentary deposits to the east and southeast.

MATERIALS AND METHODS

Soils Used

Three soil sites were selected in the Central Prairies area of Oklahoma. Each soil has a typically mature profile for the Reddish Prairies Great Soil Group. All three soils have similar topography and vegetation. Climatic environment differs mainly by the latitudinal positions of the three sites. The location of the sampling sites are shown in figure one.

Dennis Series: A sample was taken one and one/half miles northwest of Skedee, in Pawnee County. This soil has Pennsylvanian shale deposits for its parent material. The slope is 2-3 per cent and the vegetation consists of native tall grasses. The B horizon was sampled at 24 inches in depth and the C horizon at 54 inches.

Kirkland Series: A sample was taken three miles west of Edmond, in Oklahoma County. This soil has Permian clayey deposits as its parent material. The slope is 2-3 per cent and the vegetation consists of native tall grasses. The B horizon was sampled from 12-14 inches and the C horizon at 32 inches. This soil is more shallow than the usual Kirkland.

Kirkland Series: A sample was taken three miles west of Wayne, in McClain County. This soil has Permian clayey deposits as its parent material. The slope is 2-3 per cent and the vegetation consists of native tall grasses. The B horizon was sampled from 14-18 inches and the C horizon at 42 inches.

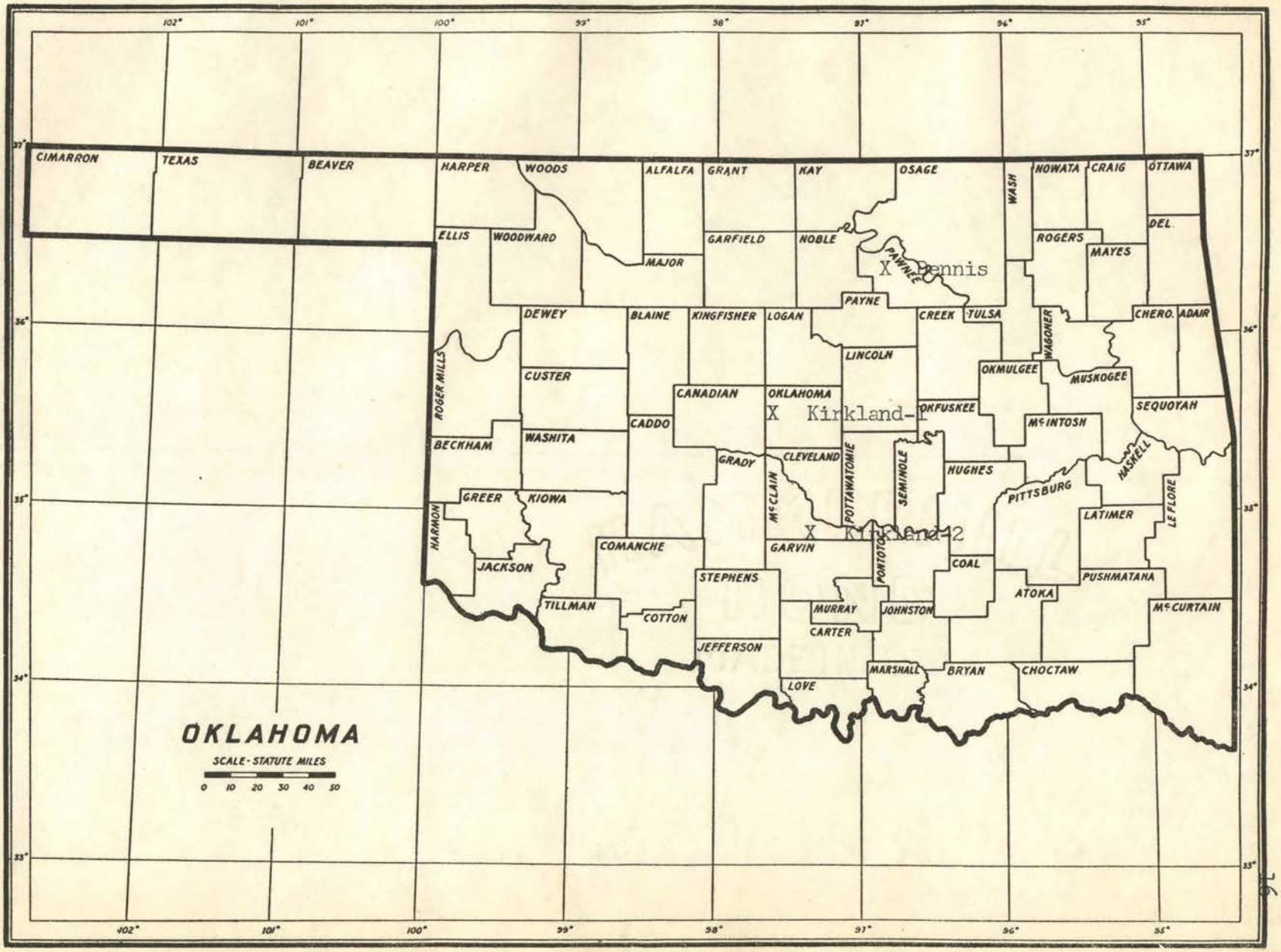

Figure 1. Location of the sampling sites

Reference Clay Minerals

The Montmorillonite was secured from The American Colloid Co., Merchandise Mart Plaza, Room 813, Chicago, Illinois as Wyoming Bentonite.

The Illite sample was secured by Dr. Charles Sarthou at the Flatwoods Outwash on Highway 15, four miles north and one-half mile east of Blue Mountain, Mississippi.

The Kaolinite was secured from United Clay Mines Corp., 109 Oakland, Trenton, New Jersey as Putnam clay.

Organic matter was removed from these clays with H_2O_2. Then the less than 2.0 micron fraction was separated by gravity sedimentation and the samples acidified with 0.05 N HCl.

Separation of the Clay-Size Fraction

A 150 gram sample of air dry soil was taken for the clay separation. The sample was divided into three 50 gram portions which were placed in beakers, and about 550 milliliters of distilled water was added. Thirty milliliters of saturated sodium oxalate solution was added to each beaker. The beakers were allowed to stand for 15 minutes, and then the contents were transferred to a dispersion cup and mechanically dispersed for 10 minutes.

After dispersion, the contents of the three beakers were transferred into a six liter carboy, and distilled water was added to bring the volume to four liters. The soil suspension was mixed and then left undisturbed for sedimentation. Stoke's law was used to calculate settling depth of five micron sedi-

ments. At the prescribed time the sediments less than five microns in diameter were drawn off to the calculated settling depth. This was usually 33 minutes per 5 cm. of settling depth. The carboy was refilled with distilled water, and the separation was repeated until the liquid in the carboy became clear.

Preparation of the Clay Sample for Fractionation in the Supercentrifuge

The sediments drawn from the carboy were flocculated with NaCl and the excess liquid drawn off. The flocculated material was transferred to a large evaporating dish, and H_2O_2 was added to destroy the organic matter. After oxidation of the organic matter, the sample was placed on a steam plate, evaporated to near dryness, and transferred to 100 ml. centrifuge tubes with approximately 5 grams of the sample per tube.

Removal of Iron Coatings: The iron coatings were removed from the clay particles with a modification of the Jefferies method (22).

Fifty Milliliters of saturated sodium oxalate was added to each centrifuge tube, and the tubes were placed in a water bath and heated to 80°C. After this temperature was reached, 10 ml. of oxalic acid solution containing 0.95 grams oxalic acid was added to each tube. The sample was stirred and heated to 90°C. A strip of magnesium ribbon, approximately 8 inches long, was placed in the hot suspension. The temperature was maintained between 90°C and 95°C., and the sample was stirred frequently. After 3-5 minutes the magnesium ribbon was removed, and an additional 5 ml. of the oxalic acid

solution added. Heating was continued for a few minutes or until the clay appeared grayish in color.

The samples were centrifuged at once and washed free of iron with 50 ml. portions of 5 per cent NaCl solution. Four washings were usually sufficient. The NaCl was removed with two washings of 80 per cent ethyl alcohol. The alcohol was removed with two washings of acetone. The excess acetone was evaporated off the sample on a steam plate.

Fractionation of the Clay Sample

The above sediments were fractionated into the following four fractions: 2.0-0.6, 0.6-0.1, and less than 0.1 micron. The 5-2.0 micron fraction was discarded due to insufficient sample.

After the excess acetone was evaporated off, the sample in the centrifuge tubes was transferred to a dispersion cup. Five milliliters of 1 N NaOH was added, and the sample was mechanically dispersed for 10 minutes. After dispersion, the suspension was poured into a 18 liter carboy, and distilled water was added to make a suspension of approximately one per cent. To remove the colloid less than 0.1 micron in diameter, the suspension was passed through a supercentrifuge at the rate of 382 ml. per minute (25°C., Specific gravity: 2.20) with a centrifugal speed of 50,000 RPM [1].

The material greater than 0.1 microns collected on a plastic sleeve inside the centrifuge bowl. This sleeve was removed

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[1] Information obtained from Dr. Charles Sarthou, Professor of Soils, Oklahoma A. and M. College.

and the clay material was transferred to a dispersion cup.

The process was repeated three times which gave a total of four runs through the centrifuge to remove the less than 0.1 micron fraction. This is in accordance with Jackson, et al (20) who found that four or five separations are usually required to separate the 0.08 micron fraction in the supercentrifuge.

The fraction greater than 0.1 micron was dispersed as before, and the suspension made to approximately 0.5 per cent. To remove the greater than 0.6 micron colloid, the suspension was passed through the supercentrifuge at the rate of 456 ml. per minute (25°C., Sp G: 2.50) with a centrifugal speed of 12,000 RPM. The material greater than 0.6 micron was again dispersed and run through the centrifuge to make a total of two times to remove the 0.6-0.1 micron fraction. (The calculations were originally made for a 0.5 micron breaking point, but later examination of the 0.5-0.1 micron fraction under the microscope showed particles greater than 0.5 microns in diameter. Consequently, 0.6 microns was considered as the upper limit of this fraction).

The fraction greater than 0.6 micron was dispersed and the 2.0-0.6 micron fraction was separated by gravity sedimentation calculated from Stoke's law. This separation was repeated until the liquid became clear. Two or three separations were usually required.

The Sharples supercentrifuge has the following bowl dimensions 1/.

Vertical distance of wall ——— 20 cm.
Radius of air column———0.734 cm.
Radius of centrifuge bowl ———2.221 cm.

Additional calculated data on the centrifuge bowl:

Volume of bowl----------309.94 ml.

Volume of air space------- 33.85 ml.

Volume of liquid----------276.09 ml.

The formula used to determine the velocity of particles toward the periphery of the bowl as given by Whitt and Baver (41) is as follows:

$$V = \frac{2r^2 k_1 Fo\ g(d_1-d_2)}{9N}$$

V is the velocity of particles toward the periphery of the bowl (cm/sec.).

r - radius of particles (cm.).

K_1 - parts per 100 of suspended material (one per cent equals one)

Fo - factor multiplying gravity.

g - acceleration due to gravity (cm/sec^2).

d_1 - density of particles (gm/cm^3).

d_2 - density of medium (gm/cm^3).

N - Viscosity of suspension (poises).

Fo g is calculated from the following formula:

$$Fog = r\ w^2$$

Fo g - centrifugal force.

r - radius of the bowl (cm).

w - angular velocity in radians per second. (5,236 rad/sec. for 50,000 RPM.).

Twenty pounds of steam pressure was needed to get a centrifugal speed of 50,000 RPM, which is also the maximum speed for this particular centrifuge. Ten pounds of steam pressure produced approximately

36,000 RPM. Two or three pounds pressure was sufficient for 12,000 RPM, which was used for the 0.6 micron separation.

Hydrogen Saturation of the Colloid Fractions

The three colloid size separates were in suspensions after being fractionated by the supercentrifuge. NaCl was added to flocculate the suspension, and the flocculated material was poured into a large evaporating dish. The sample was evaporated to near dryness on a steam plate, transferred to 100 ml. centrifuge tubes, and acidified with 0.05 N HCl. Approximately 50 ml. of acid was added to each centrifuge tube. The suspension was stirred; then centrifuged to settle the sample. The liquid was decanted and the process repeated until the exchange capacity of the colloid was saturated with hydrogen. This usually took 6-7 washings with acid. Sufficient washing was indicated when the normality of the acid was not reduced by the sample.

The sample was washed twice with 80% ethyl alcohol and twice with acetone. The excess acetone was removed by evaporation. After drying, the sample was crushed to pass a 100 mesh sieve and stored in stoppered test tubes.

Determination of Base Exchange Capacity

The base exchange capacity was determined by the $BaCl_2$ method as used by Whit and Baver (41).

Approximately 0.5 gram of oven dry H-colloid sample was weighed, and placed in a 100 ml. centrifuge tube. Approximately 60 ml. of neutral 1 N $BaCl_2$ solution was added. The sample was stirred and allowed to stand overnight. The sample was stirred again and then cen-

trifuged. The wash-solution was decanted into a 250 ml. erlinmeyer flask, and two drops of phenolphthalein indicator was added. The solution was then titrated with 0.0500 N NaOH until a slight pink color persisted for 30 seconds. The process was repeated (except subsequent washes did not stand overnight) until the titration with NaOH became constant, which varied from 2-5 washings depending on the exchange capacity.

The formula used to determine base exchange capacity is as follows:

$$E = \frac{(w_1 - W_2 - \ldots W_n)\ (N)\ (100)}{S}$$

E - base exchange capacity (m.e./100 grams).

w - milliliters NaOH for each washing.

N - normality of the NaOH.

S - sample weight in grams.

Ethylene Glycol Retention

The method used for the glycol retention determinations was taken from Dyal and Hendricks (11,12).

Approximately 1.1 grams of the H-colloid was placed in a tared aluminum weighing dish, with a diameter of 5 centimeters, oven dried at 110°C. and then weighed. Another sample of approximately 1.2 grams was placed in a porcelain evaporating dish, held at 600°C. for 4 hours in a muffle furnace, cooled in a desiccator, transferred to a tared aluminum weighing dish, and weighed.

After the samples were weighed, about 1.0 ml. of redistilled ethylene glycol was added to each one and the samples were set aside

overnight to allow uniform wetting. They were then placed in a vacuum desiccator above 500 grams of anhydrous $CaCl_2$, and the desiccator was evacuated to a vacuum of 2 mm. of mercury. After 24 hours of distillation, dry air was introduced into the desiccator to remove the vacuum. The samples were removed and weighed.

Dyal and Hendricks (11) recommended less than 0.1 mm. mercury vacuum, but it could not be attained in this work. Performance of the system can be tested by evaporation of ethylene glycol at the rate of 1.0 ml. in 8 hours (11). However, more than 3.0 ml. of ethylene glycol was evaporated in 24 hours in this investigation.

Non-exchangeable Potassium

The method used to determine non-exchangeable potassium was a modification of the method found in A.O.A.C. Methods of Analysis, Sixth Edition, 1945, page 10.

One gram of the hydrogen saturated clay sample was mixed thoroughly with one gram of dry NH_4Cl and 8 grams of $CaCO_3$ in an agate mortar. The mixture was transferred to a platinum crucible. The crucible was heated gradually until fumes of NH_4 salts no longer appeared. The lower 3/4 of the crucible was brought to red heat and maintained at this temperature for 60 minutes.

The fused mass was transferred to a porcelain dish, slaked with hot water, and ground thoroughly with an agate pestle. The fusion was washed 5 times by decantation with hot water, transferred to a filter and washed with an additional 300 ml. of hot water. Calcium was precipitated in the filtrate with $(NH_4)_2CO_3$ solution. The filtrate with the precipitated calcium was transferred to a filter and washed with water. The solution was evaporated to approximately 50

ml. and then transferred to a 100 ml. volumetric flask. Ten milliliters of a 1,000 ppm lithium stock solution was added. The flask was brought up to volume with water. Potassium was determined on a flame photometer and the results reported as per cent K_2O.

Mechanical Analysis

A 20.00 gram sample of the original soil was placed in a 200 ml. nursing bottle containing 150 ml. of distilled water. Ten milliliters of sodium hexametaphosphate solution was added as a dispersing agent 2/, and the bottle was placed in a mechanical shaker and shaken overnight. The sample was then washed through a 300 mesh sieve, which retained the sand. The sand was washed into an evaporating dish, dried, and weighed. The remainder of the sample was placed in a dispersion cup and a tablespoon of coarse sand was added to it. The sample was then dispersed for 25 minutes with a mechanical mixer (38).

After dispersion, the sample was poured into a liter graduated cylinder and distilled water added to bring the volume to one liter. The suspension was stirred for 30 seconds and two 40 ml. aliquots were immediately pipetted into 50 ml. centrifuge tubes. The suspension in the cylinder was allowed to stand for a 2 micron determination as calculated by Stoke's law. Density was assumed to be 2.61 grams/cm^3.

$$V = \frac{2/9\ g\ (d - d_p)\ r^2}{n}$$

2/Sodium hexametaphosphate$(NaPO_3)_6$ solution was made by dissolving 35.70 grams of commercial "Calgon" and 7.94 grams of Na_2CO_3 in water and bringing the volume to one liter (40).

V - velocity of fall (cm/sec^2).

g - acceleration due to gravity (cm/sec^2).

d_p - density of the particle (gms/cm^3).

d - density of the liquid (gms/cm^3).

n - viscosity of the liquid (poises).

r - radius of the particle (cm.).

At the end of the calculated time, a 25 ml. automatic pipette (calibrated for full volume) was lowered into the suspension at the prescribed depth, and the pipette was slowly filled (40 sec.) with light suction. The pipetted suspension was emptied into a weighed evaporating dish, dried in an oven overnight, and weighed on an analytical balance.

The 40 ml. aliquots of the original suspension were centrifuged for a determination of 0.6 micron and 0.1 micron size particles. Calculations were made from the formula of Steele and Bradfield (37).

$$t = \frac{n \log \frac{R_2}{R_1}}{3.81\, N^2\, r^2\, (d_p - d)}$$

t - time in seconds

n - viscosity in poises.

R_2 - distance in cm. that the particle will be from the center of rotation at the end of t.

N - Number of revolutions per second.

r - radius of the particle in cm.

d_p - density of the particle in $grams/cm^3$.

d - density of the liquid in $grams/cm^3$.

R_1 - distance in cm. that the particle is from the center of rotation at the beginning of t.

Particle density was assumed to be 2.50 and 2.20 grams/cm^3 for 0.6 micron and 0.1 micron respectively (39). Time was counted from when the centrifuge switch was turned on to when it was turned off.

After the sample was centrifuged, it was taken from the centrifuge and placed in a test tube rack. A 5 ml. pipette (calibrated for full volume) was lowered R_2-R_1 distance into the suspension. The pipette was filled slowly with the aid of gentle suction supplied by vacuum from a siphon bottle. The pipetted suspension was emptied into a tared evaporating dish. The pipette was washed twice with distilled water, and these washings were added to the suspension in the evaporating dish. The pipette was dried with acetone and made ready for the next sample. Only two samples were centrifuged at a time.

Calculations are as follows:

$$\text{Per cent sand} = \frac{(\text{weight of sand})\ (100)}{\text{weight of oven dry sample}}$$

$$\text{Per cent silt} = 100 \text{ minus per cent sand and clay}$$

$$\text{Per cent clay} = \frac{(V)\ (C-D)\ (100)}{(v)\ (S)}$$

V - volume of suspension in ml.

C - weight of clay in evaporating dish in grams.

D - weight of the dispersing agent in grams.

v - volume of the pipette in ml.

S - oven dry sample weight in grams.

RESULTS AND DISCUSSION

The soil samples shall be referred to by their series name. See Figure 1.

Dennis	Pawnee County
Kirkland-1 or Kirk-1	Oklahoma County
Kirkland-2 or Kirk-2	McClain County

The horizons shall be listed with the usual lettering system.

pH

The pH of the soil samples was determined with a Beckman glass electrode pH meter. The soil paste method was used. Results are given in Table 2.

Table 2. pH of the soil samples.

Soil	Horizon	pH
Dennis	B	6.0
	C	7.7
Kirkland-1	B	6.7
	C	8.0
Kirkland-2	B	6.8
	C	7.6

The B horizon of all three soils are slightly acid and the C horizons alkaline in reaction.

Mechanical Analysis

The supercentrifuge is not practical for the quantitative separation of the clay fraction in soils. Mechanical analysis by the pipette method was used to determine the percentages of the three size separations that were fractionated.

The results of the Mechanical Analysis are shown in Tables 3 and 4.

All the samples were high in clay, and, except for the C horizon of the Kirkland-1, all had a low sand content and medium to high percentage of silt.

The mechanical analyses of the clay fractions show a high proportion of colloid less than 0.1 micron in diameter. The Kirkland-2 horizon was the only sample that had a 0.6-0.1 micron separation larger than the fraction less than 0.1 microns. The Dennis series, which has Pennsylvanian shale deposits for its parent material, had the highest per cent of fine clay and the lowest per cent of coarse clay, 2.0-0.6 microns, as compared to the Kirkland series which developed from Permian clayey deposits.

The B horizons of the three soils were lower in the 2.0-0.6 and 0.6-0.1 micron clay fractions and higher in the fraction less than 0.1 micron, as compared to their respective C horizons. The Dennis C contained 6.2 per cent less of the fine clay fraction than the B horizon. The two C horizons of Kirkland were 9.4 and 31.2 per cent less than their respective B horizons in the same fraction.

Sodium hexametaphosphate was found to be superior to a combination of sodium oxalate, sodium hydroxide, and sodium silicate for a dispersing agent. Dispersion with the use of the sodium hexametaphosphate increased the fine clay fraction, by approximately 25 per cent.

Mechanical agitation by a soil dispersion mixer was found to be essential for a more complete dispersion of the clay, especially for the finer fractions. Dispersion for 25 minutes by a soil dispersion mixer increased the fine clay fraction by approximately 80 per cent as compared to dispersion in a mechanical shaker for 14 hours.

Table 3. Mechanical analysis of the soil samples.

Soil	Per cent Sand >50 microns	Per cent Silt 50-2.0 microns	Per cent clay 2.0-0.6 microns	Per cent clay 0.6-0.1 microns	Per cent clay <0.1 microns	Per cent Total Clay	Texture
Dennis B	13.9	44.0	1.2	14.0	26.9	42.1	SiC
C	23.2	37.1	3.0	13.8	22.9	39.7	CL
Kirk-1 B	21.9	30.9	3.6	17.0	26.6	47.2	C
C	41.8	19.5	4.2	16.3	18.2	38.7	CL
Kirk-2 B	16.5	46.5	2.8	10.8	23.4	37.0	SiCL
C	20.4	44.0	2.9	21.3	11.4	35.6	CL

Table 4. Mechanical analysis of the clay fraction (total clay fraction as 100 per cent)

Soil	Coarse Fraction Per cent clay 2.0-0.6 Microns	Medium fraction Per cent clay 0.6-0.1 Microns	Fine Fraction Per cent clay <0.1 Microns
Dennis B	2.9	33.2	63.9
C	7.6	34.7	57.7
Kirk-1 B	7.6	36.0	56.4
C	10.9	42.1	47.0
Kirk-2 B	7.6	29.2	63.2
C	8.2	59.8	32.0

Base Exchange Capacity

Base exchange capacity was determined to find indications of predominant clay minerals in the three clay fractions.

The three soils were found to have similar base exchange capacities, and little difference was found between the B and C horizons (Table 5).

Exchange capacity increased markedly with a decrease in particle size of the clay fractions. The exchange capacity of the fine clay fraction averaged approximately 1½ times greater than the 0.6-0.1 fraction and 3 times greater than the 2.0-0.6 micron fraction.

Increase of exchange capacity with a decrease in particle size may be attributed to three factors:

(a) The error of omission in the coarser fractions would produce a lower apparent exchange capacity. As pointed out in the review of literature, quartz, hydrated iron oxides and other impurities are found in clay size fractions (10,18,24,25). Quartz may be a dominant impurity in the coarser fractions. Most, but not all, of the iron oxides were removed from the clay in this investigation by the oxalic acid-magnesium method (21). The two Kirkland soils were considered to be high in this oxide. Observation of the reddish color in the three size fractions would indicate that more iron oxide occurred in the 0.6-0.1 micron fraction than the other two fractions, although the fraction less than 0.1 micron showed considerable amounts. Incomplete removal of the iron oxides allowed this observation.

(b) The fractionation of the clay fraction tend to separate the different clay minerals (2, 10, 30, 35). Montmorillonite with a high base exchange capacity is usually found in the fractions less than 0.1 micron

Table 5. Base exchange capacities of the clay fractions and the reference clay minerals. m.e./100 grams

Soil	Fraction 2.0-0.6 Microns	Fraction 0.6-0.1 Microns	Fraction < 0.1 Microns	Calculated for Total Clay	Calculated for Total Soil*
Dennis B	14.8	33.3	57.1	48.0	20.2
C	15.8	34.6	55.0	44.9	17.8
Kirk-1 B	20.4	37.0	54.2	45.4	21.4
C	21.9	38.4	54.5	44.2	17.1
Kirk-2 B	17.2	37.3	56.4	47.8	17.7
C	14.7	35.6	51.3	38.9	13.8

Clay Minerals Mineral	< 2.0 Microns
Montmorillonite	79.9
Illite	37.1
Kaolinite	3.4

*Assuming no exchange capacity in the sand and silt fractions.

in diameter. Illite and Kaolinite with lower exchange capacities tend to be found in the coarser clay fractions.

(c) The particle surface area available for base exchange capacity is considerable less in the coarser clay fractions. For example, Kaolinite has a total surface area of approximately 28 m^2/gram while Montmorillonite has approximately 810 m^2/gram (12).

The base exchange capacity of the reference dlay minerals are within the ranges given in literature. The Montmorillonite may be considered somewhat low. The Illite has a high exchange capacity and the 3.4 m.e./100 grams for Kaolinite may be considered a little low.

Non-exchangeable Potassium

Non-exchangeable potassium was determined to indicate the extent of Illite type clays.

The results show considerable quantities of Illite in the clay fractions as indicated by the K_2O percentages (Table 6). One trend is evident. The 0.6-0.1 micron fraction usually had the highest K_2O content of the three size fractions. The fraction less than 0.1 midrons usually had the least. This is in accordance with Pennington and Jackson who found that Illite is more prominent in the coarser clay fractions (30).

The B and C horizons show some differences in K_2O content. Data show that the C horizon has the higher per cent of K_2O . Assuming 6 per cent K_2O as reference for pure Illite, the 0.6-0.1 micron fractions have from approximately 35-40% Illite, the 2.0-0.6 fractions approximately 15 - 30%, and the less than 0.1 micron fractions approximately 15 - 25%.

Six per cent K_2O for pure Illite was used due to Gieseking's (13)

Table 6. Non-exchangeable Potassium of the clay fractions and the reference clay minerals (as per cent K_2O)

Soil	Fraction 2.0-0.6	Fraction 0.6-0.1	Fraction <0.1	Total Clay*
Dennis B	1.01	2.16	1.02	1.40
C	1.85	1.98	1.02	1.42
Kirk-1 B	1.25	2.41	0.90	1.47
C	1.83	2.39	1.78	2.04
Kirk-2 B	1.82	2.05	0.86	1.28
C	1.40	2.31	0.98	1.81

Reference Clay Minerals

Montmorillonite	0.24
Illite	3.42
Kaolinite	0.02

*Calculated

statement that most workers assume this value for Illite. The value found for the reference Illite sample in this investigation was considered too low to be used.

Ethylene Glycol Retention

Ethylene glycol retention in clays was used as an indication of Montmorillonite and the results obtained are presented in Table 7.

Glycol retention of the unheated samples increased with a decrease in particle size. The 2.0-0.6 micron fractions averaged approximately 30 mgm/gm sample of glycol retention; 0.6-0.1 micron fractions, 80 mgm/gm; and the fractions less than 0.1 micron, 130 mgm/gm.

Table 7. Ethylene Glycol Retention of the clay fractions and the reference clay minerals. mgm/gram sample.

Soil	Clay Fraction	Treatment None	Treatment Heat	Diff.	% Mont.
Dennis B	2.0-0.6	29	13	16	8.6
	0.6-0.1	94	17	77	41.2
	<0.1	131	51	80	42.8
C	2.0-0.6	38	13	25	13.4
	0.6-0.1	78	23	55	29.4
	<0.1	151	32	119	63.6
Kirk-1 B	2.0-0.6	38	14	24	12.8
	0.6-0.1	74	28	46	24.6
	<0.1	147	59	88	47.0
C	2.0-0.6	47	19	28	15.0
	0.6-0.1	100	25	75	40.1
	<0.1	125	34	91	48.7
Kirk-2 B	2.0-0.6	37	12	25	13.4
	0.6-0.1	81	25	56	30.0
	<0.1	157	44	113	60.4
C	2.0-0.6	33	10	23	12.3
	0.6-0.1	95	33	62	33.2
	<0.1	145	35	110	58.8
Montmorillonite		205	18	187	100.0
Illite		97	41	56	30.0
Kaolinite		19	10	9	-

Heating the sample at 600°C for four hours caused a considerable reduction in the ability of the samples to retain ethylene glycol. This is due to the destruction of interlayer swelling of Montmorillonite and, to some extent, of Illite in the fraction.

The reference Montmorillonite showed the greatest reduction of glycol retention with heating, and this reduction was used for the calculation of Montmorillonite in the clay fradtions. The Illite sample showed a higher interlayer glycol retention than usually accorded to this mineral. The Kaolinite is typical with a very low retention in both the heated and unheated treatments.

Glycol retention found in this investigation for Montmorillonite was considerably lower than the value used by Dyal and Hendricks (11,12). They found Montmorillonite to retain 240-290 mgm/gm clay of ethylene glycol. Possible explanation of the low retention may be due to high room temperatures encountered during the distillation of the glycol-clay in the vacuum desiccator. Temperatures varied from 30-34°C. due to a hot month of August. High temperatures would hasten the distillation of the glycol from the clay. A few samples were checked at various periods of distillation and mgm/gm retention plotted against the hours of distillation (Figure 2). These curves show that a shorter distillation time could have been used. The error due to 24 hours of distillation probably would indicate a higher per cent of Montmorillonite, especially in the coarser clay fractions, than actually exist. The curves show that the coarser clay fractions reached a more or less equilibrium point before the fine fractions.

After examination of the glycol-clay distillation curves, corrections were made to partly compensate for the long distillation time used. The

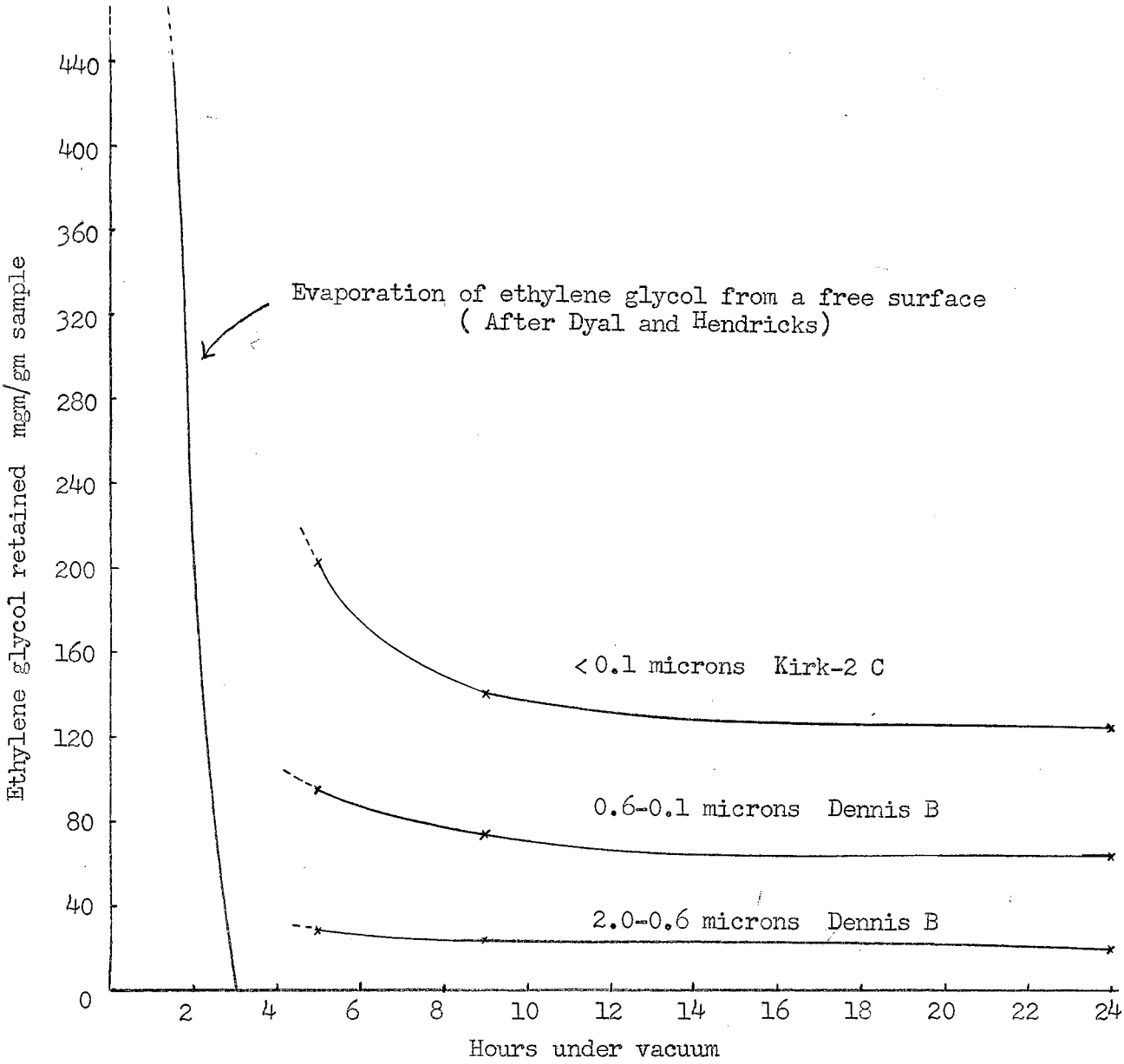

Figure 2. Comparison of the ethylene glycol retention curves of the three size fractions of the clay

corrections were based on the curves shown in Figure 2. The points on the curves at five hours of distillation was used for the 2.0-0.6 micron unheated fractions and all of the heated samples. A correction of 5 mgm. was added to these samples. Similarly, a reading at 9 hours of distillation was used for the 0.6-0.1 and the less than 0.1 micron fractions with a correction of 10 and 20 mgm. of glycol retention, respectively. A distillation period of 14 hours was considered adequate for Montmorillonite and 20 mgm. of glycol retention was added as a correction factor.

The mica-like clay minerals, according to Dyal and Hendricks (11), retain approximately 40-70 mgm/gm of glycol when unheated and approximately 1-30% less when heated. The Illite sample used in this investigation showed a high amount of interlayer swelling which indicates the presence of Montmorillonite as an impurity. Evidence of this impurity is supported by a high base exchange capacity and a low per cent of non-exchangeable potassium.

Dyal and Hendricks (11) found Kaolinite to retain 14 and 7 mgm/gm of glycol retention, unheated and heated respectively.

Correlation of the Tests

The various tests performed with the clay fractions should point toward the possibility of estimating percentages of Illite and Montmorillonite in these fractions. No tests were performed to explicitly indicate Kaolinite. The presence of Kaolinite in the three soils was considered to be low.

Estimation of the approximate content of Illite and Montmorillonite was made on the basis of the following assumptions: (a) the entire non-exchangeable potassium content is present in the clay mineral Illite:

(b) Illite contains an average of 6% K_2O; (c) ethylene glycol retention of the internal surface of clays is attributed mainly to Montmorillonite; (d) 187 mgm/gm internal surface glycol retention is considered to represent pure Montmorillontie; and (e) the base exchange capacities of Montmorillonite and Illite are 80 and 25 m.e./100 grams, respectively.

Calculations based on these assumptions are given in Table 9.

Correlation of K_2O content, glycol retention, and base exchange capacity shows the inherent dangers of applying an ideal situation to soils. However, most of the calculated base exchange capacities (Table 9) do agree reasonably close to the experimental values. The calculated exchange capacities were based on the percentages of Montmorillonite and Illite computed to be present in the fraction. The largest errors were found in the fine clay fractions of the Dennis B and the Kirkland-1 B horizons. These were the two fractions that were considered to have a low glycol retention.

After the percentages of the Montmorillonite and Illite were computed, the clay fractions still contained a considerable amount that was left unaccounted for. This was especially true for the 2.0-0.6 micron fractions (Table 8) which had an average of 55-75% that was unknown.

Impurities were believed to comprise most of the left-over portion of the fraction. Quartz would probably be the dominant impurity in the coarse clay fractions and iron oxides in the finer fractions. Since no determinations were made for Kaolinite, it would be included in the unaccounted portion of the clay fraction.

Analysis of the Individual Soil

Dennis: The clay fraction of the B horizon was estimated to con-

Table 8. Estimation of the percentages of Montmorillonite and Illite in the Clay fractions.

Soil	Fraction 2.0-0.6	Fraction 0.6-0.1	Fraction 0.1	Total Clay Fraction (calculated)
		% Montmorillonite		
Dennis B	8.6	41.2	42.8	41.3
C	13.4	29.4	63.6	47.9
Kirk-1 B	12.8	24.6	47.0	36.3
C	15.0	40.1	48.7	41.4
Kirk-2 B	13.4	30.0	60.4	48.0
C	12.3	33.2	58.8	39.7
		% Illite		
Dennis B	16.8	36.9	17.0	23.6
C	30.8	33.0	17.0	23.6
Kirk-1 B	20.8	40.2	15.0	24.5
C	30.5	39.8	29.7	34.0
Kirk-2 B	30.3	34.2	14.3	21.3
C	23.3	38.5	16.3	30.1
		% Unaccounted for		
Dennis B	74.6	21.9	40.1	35.1
C	55.8	37.6	19.4	28.5
Kirk-1 B	66.4	35.2	38.0	39.2
C	54.5	20.1	21.6	24.6
Kirk-2 B	56.3	35.8	25.3	30.7
C	64.4	28.3	24.9	30.2

Table 9. Comparison of calculated base exchange capacity to experimentally determined base exchange capacity.* m.e./100 grams

Fraction		Soil					
		Dennis		Kirkland-1		Kirkland-2	
		B	C	B	C	B	C
2.0-0.6	Exp	14.8	15.8	20.4	21.9	17.2	14.7
	Calc	11.0	18.4	15.4	19.6	18.3	15.7
	Dif	3.8	-2.6	5.0	2.3	-1.1	-1.0
0.6-0.1	Exp	33.3	34.6	34.6	38.4	37.3	35.6
	Calc	42.4	31.8	31.8	42.0	32.6	36.2
	Dif	-9.1	2.8	2.8	-3.6	4.7	-0.6
< 0.1	Exp	57.1	55.0	54.2	54.5	56.4	51.3
	Calc	38.5	55.1	41.4	46.4	51.8	51.1
	Dif	18.6	-0.1	12.8	8.1	4.6	0.2
Total Clay	Exp	48.0	44.9	45.4	44.2	47.8	38.9
	Calc	38.9	44.2	35.2	41.6	43.6	39.3
	Dif	9.1	0.7	10.2	2.6	4.2	-0.4

*Calculated on basis: Montmorillonite 80 m.e./100 grams
Illite 25 m.e./100 grams

tain approximately 40% Montmorillonite and 20% Illite. The Montmorillonite per cent is thought to be low due to experimental error. Evidence from the calculated and experimental data point toward an error.

The clay fraction in the C horizon was estimated to contain approximately 50% Montmorillonite and 20% Illite.

The percentages in tabular form are given in Table 10.

Table 10. Estimation of Montmorillonite and Illite in the clay fraction of the Dennis soil

	% Montmorillonite	% Illite	% Uaccounted for
B Horizon	40 (50)*	20	40 (30)*
C Horizon	50	20	30

*Considered to be more nearly correct.

The results indicate that little difference exists in the clay mineralogical composition between the B and C horizons of this particular soil. Base exchange capacity, K_2O content, ethylene glycol retention (except the fine fraction of the B horizon), and mechanical analysis of the clay fraction are reasonably similar for the two horizons.

The results of the various analysis for this soil are illustrated in Figrues 3,4,5 and 6.

Mechanical analysis of the total sample shows that the B horizon contains more clay and less sand due to possible eluviation of fine particles from the A horizon. pH of 6.0 indicates that a significant amount of leaching has taken place in the B horizon. The C horizon is alkaline with a pH of 7.7.

The Dennis series is considered a soil with a mature profile. However, the weathering influence upon the soil particles in the B horizon is not sufficiently great to be significantly detected by the tests used

DENNIS SOIL

Illustration of the various analyses with comparison of the size fractions and the B and C horizons

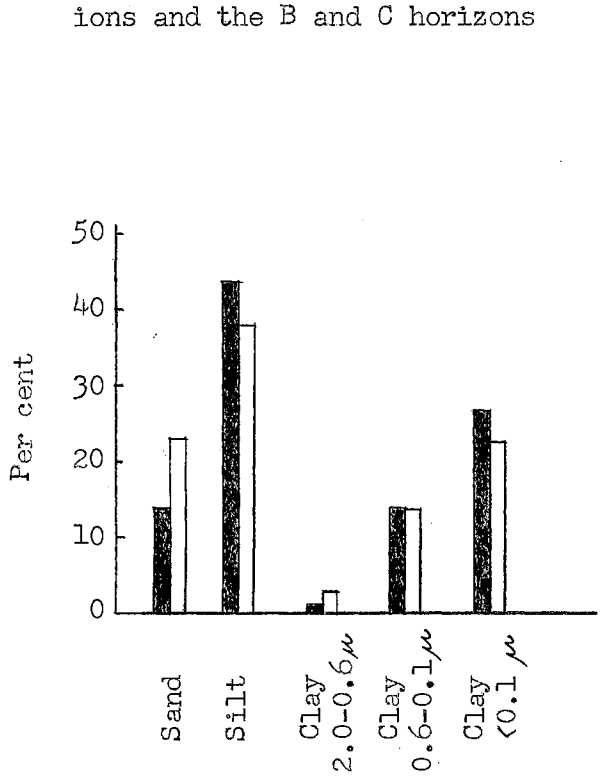

Fig. 3 Mechanical analysis

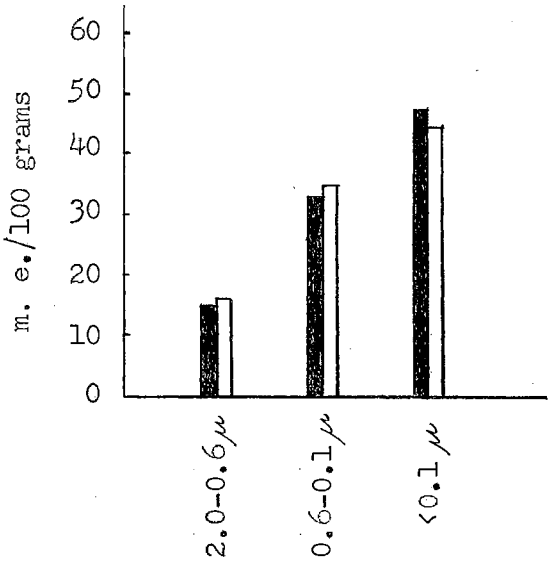

Fig. 4 Base exchange capacity

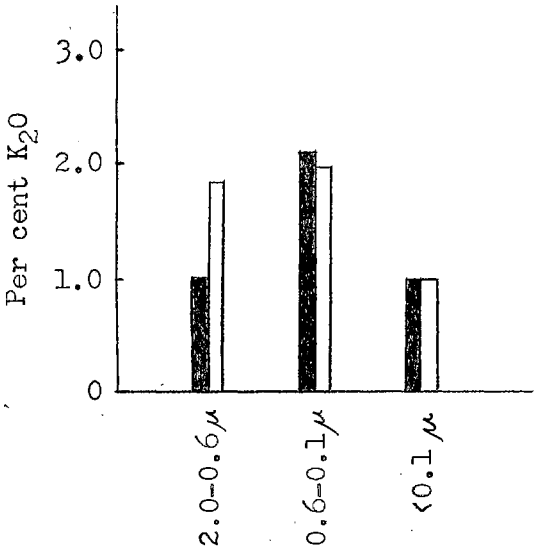

Fig. 5 Non-exchangeable potassium

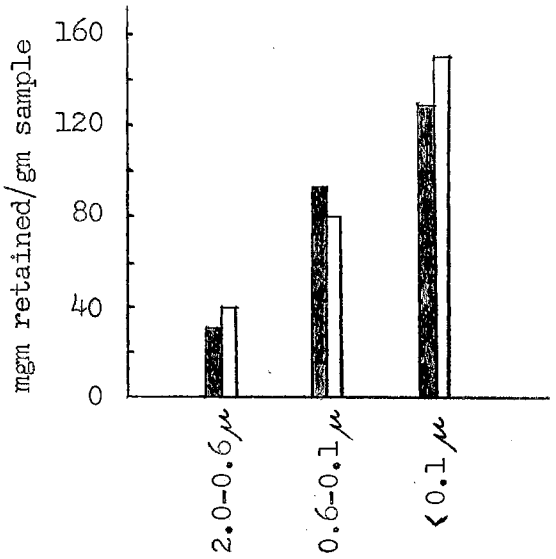

Fig. 6 Total ethylene glycol retention

in this study. Possibly, the factors of weathering (intensity, capacity, and time) as put forth by Jackson, et al (20) have not shifted the weathering stage to the right as far as might be expected. His weathering sequence shows that Illite weathers to mica intermediates then to Montmorillonite. A further study of the weathering influence on the complete soil profile would be an aid to soil morphology and classification.

Kirkland-1: The clay fraction in the B horizon was estimated to contain approximately 35% Montmorillonite and 25% Illite. The Montmorillonite per cent is suspected of being low.

The clay fraction in the C horizon was estimated to contain 40% Montmorillonite and 35% Illite.

The percentages in tabular form are given in Table 11.

Table 11. Estimation of Montmorillonite and Illite in the clay fraction of the Kirkland-1 soil.

	% Montmorillonite	% Illite	% Unaccounted for
B horizon	35 (45)*	25	40 (30)*
C horizon	40	35	25

*Considered to be more nearly correct.

This soil shows evidence of more differentiation between the B and C horizons with respect to clay composition than the Dennis soil.

Mechanical analysis shows that the B horizon contains considerably more of the finer soil particles than the C horizon.

Base exchange capacities of the B and C horizons are similar in all three clay fractions. This would tend to indicate that the percentage of Montmorillonite calculated for the B horizon was low, since the ethylene

KIRKLAND-1 SOIL

Illustration of the various analyses with comparison of the size fractions and the B and C horizons

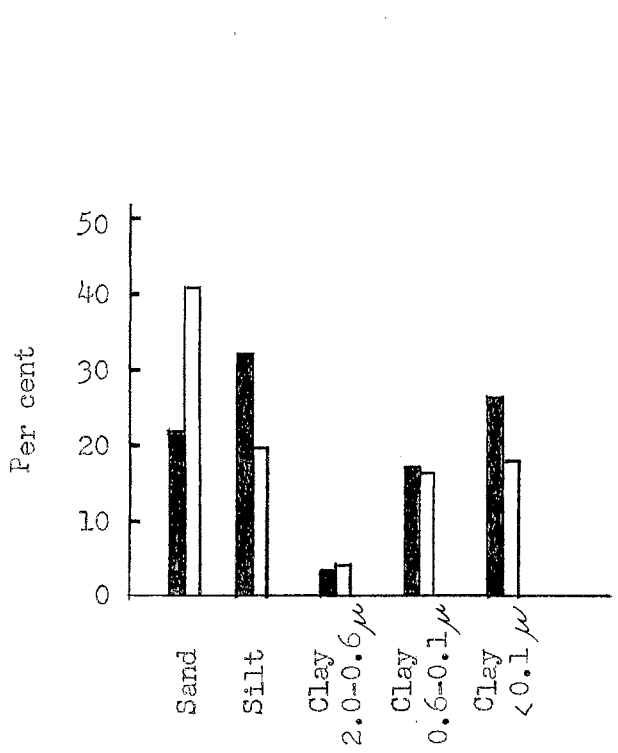

Fig. 7 Mechanical analysis

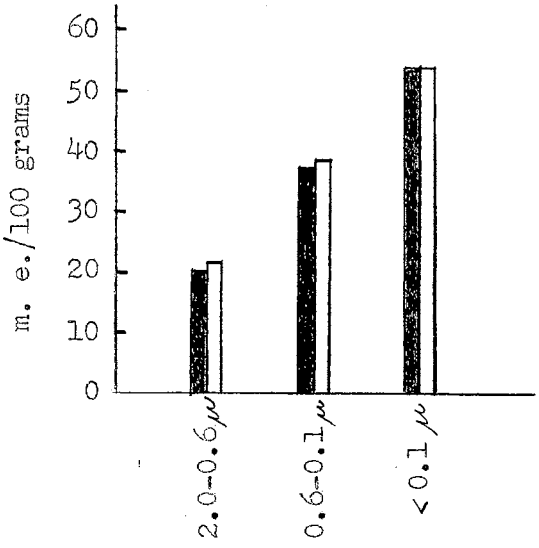

Fig. 8 Base exchange capacity

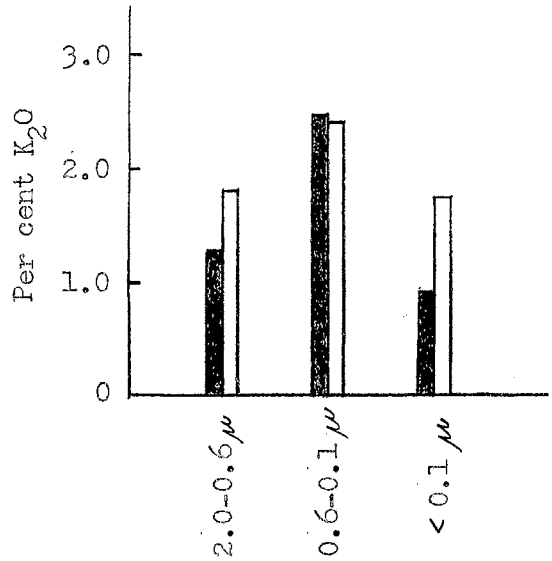

Fig. 9 Non-exchangeable potassium

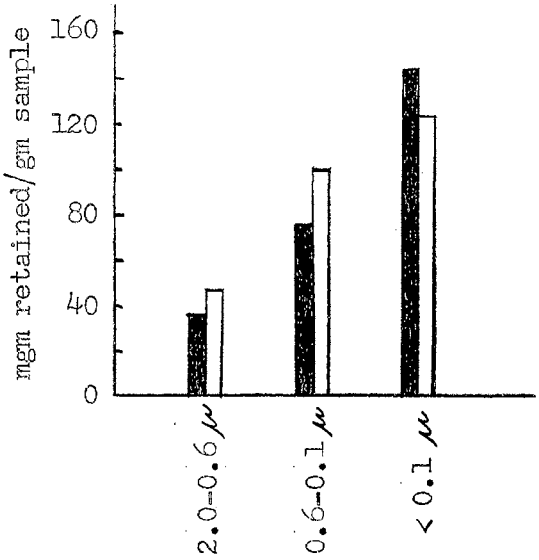

Fig. 10 Total ethylene glycol retention

glycol retention of the fine fraction was considered low. If 10% more Montmorillonite was indicated in the clay fraction of the B horizon, the calculated base exchange capacity (Table 8) would more nearly equal the experimentally determined base exchange capacity.

The Illite per cent in the clay fraction of this soil was found to be higher than what was expected. The C horizon contains more Illite than the B horizon. This would tend to indicate a possible shift to the right in weathering according to Jackson's, et al (20) scheme of weathering sequence.

This soil, as well as the other two soils, show a higher per cent of non-exchangeable potassium in the fraction, 0.6-0.1 microns in diameter than in the other two fractions (Figrues 5, 9, and 13). The fine fraction, less than 0.1 microns, has the lowest K_2O per cent.

Illustrations of the various analyses on this soil are presented in Figures 7, 8, 9 and 10.

Kirkland-2: The clay fraction of the B horizon was estimated to contain approximately 50% Montmorillonite and 20% Illite.

The clay fraction of the C horizon was estimated to contain 40% Montmorillonite and 30% Illite.

The percentages in tabular form are given in Table 12.

Table 12. Estimation of Montmorillonite and Illite in the clay fractions of the Kirkland-2 soil.

	% Montmorillonite	% Illite	% Unaccounted for
B horizon	50	20	30
C horizon	40	30	30

KIRKLAND-2 SOIL

Illustration of the various analyses with comparison of the size fractions and the B and C horizons

LEGEND

■ B horizon

□ C horizon

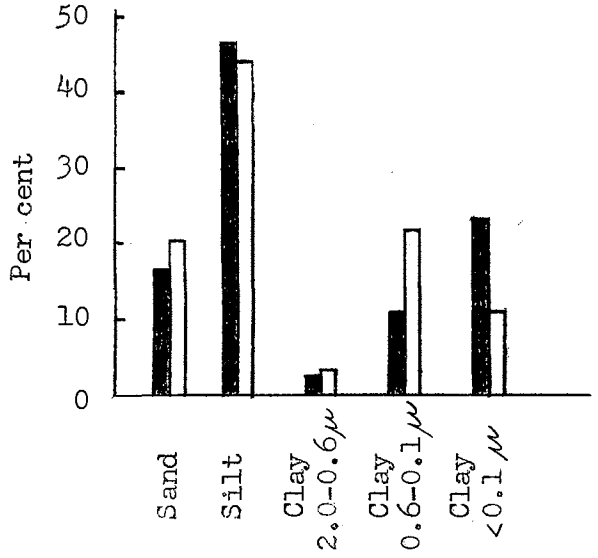

Fig. 11 Mechanical analysis

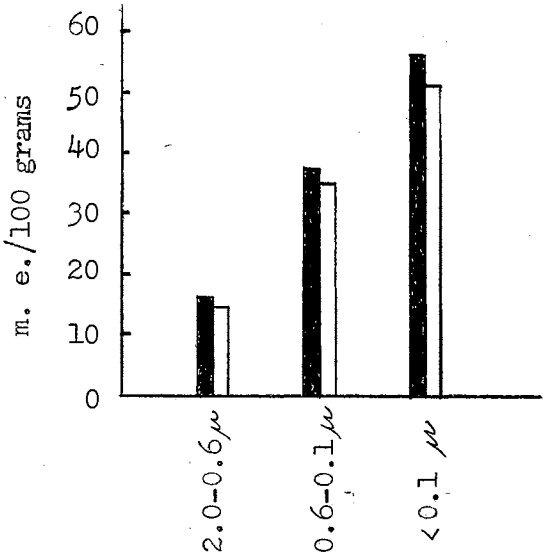

Fig. 12 Base exchange capacity

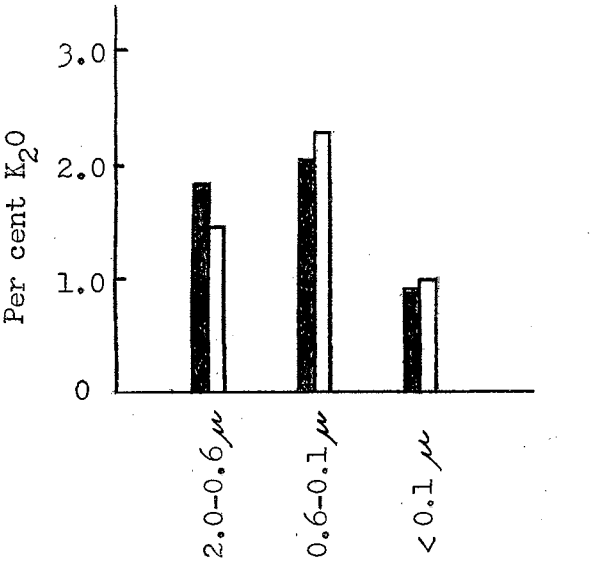

Fig. 13 Non-exchangeable potassium

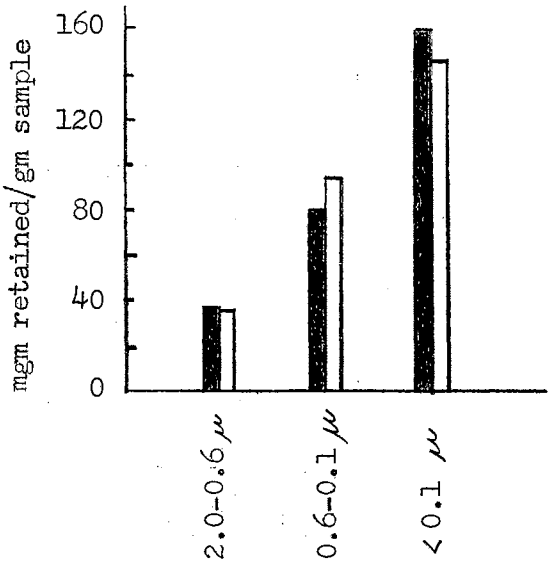

Fig. 14 Total ethylene glycol retention

The correlation of the various tests performed with the clay fractions of this soil shows closer agreement than with the two other soils. Calculated base exchange capacities agree reasonably close with the experimental values.

As with Kirkland-1, the clay fraction of the B horizon shows less Illite and more Montmorillonite than the C horizon.

Although the total clay per cent is nearly the same for the B and C horizons, a look at the mechanical analysis of the clay fraction shows that the C horizon has considerably less of the fine clay and more of the medium clay than the B horizon. This would indicate a breakdown of the soil particles within the clay-size fraction since the total clay per cent of the B horizon is only slightly higher than that of the C horizon.

CONCLUSIONS

The Dennis and the two Kirkland soils show a similar mineralogical composition. A difference due to parent material was not readily evident. Nor was there detectable evidence of differences due to the latitudinal positions of the sampling sites. This would be in accordance with Sutton's (38) conclusion that the present geological parent material and recent climatic environment have had little effect upon the type of clay minerals found in these soils.

Further study would be needed to show differences or trends in clay composition of the soils in different areas of Oklahoma. The Dennis and the two Kirkland soils may or may not be typical for the soils derived from the Pennsylvanian and Permian clayey parent materials, respectively.

If these soils are typical of their series, the results of this investigation may be summarized as follows:

Table 13. Estimation of the percentages of Montmorillonite and Illite found in the Dennis and Kirkland series.

Soil Series	Horizon	% Montmorillonite	% Illite
Dennis	B	40-50	20
	C	50	20
Kirkland	B	40-50	20-25
	C	40	30-35

Secondary Conclusions

Mechanical analysis shows that a large part of the clay was found in the less than 0.1 micron fraction. The amount of clay in the coarse fraction was very small. The less than 0.1 micron fractions usually comprised approximately one-half of the total clay.

Base exchange capacity increased markedly with a decrease in particle size.

K_2O per cent was found to be the highest in the 0.6-0.1 micron fractions.

Ethylene glycol retention increased rapidly with a decrease in particle size, an indication that Montmorillonitic type clay minerals are more predominant in the finer fractions.

The 2.0-0.6 micron fractions contained a large per cent of impurities, which was probably quartz.

Results indicate that Montmorillonite and Illite clay minerals tend to segregate into different particle size ranges.

SUMMARY

The clay fraction of the B and C horizons of a Dennis and two Kirkland soils were analyzed for the percentages of Montmorillonite and Illite.

The scheme of analysis consisted of the fractionation of the clay-size material of the soil into three fractions. Ethylene glycol retention, non-exchangeable potassium, and base exchange capacity determinations were made on the three fractions. Percentages of Montmorillonite and Illite were computed from the evidence of these determinations.

Mechanical analysis of the clay-size material gave the per cent that each fraction was of the whole. This per cent was multiplied times the computed per cent of Montmorillonite or Illite in that fraction. From these computations the content of the two clay minerals was determined for the total clay fraction.

The clay fraction of the Dennis B horizon was estimated to contain 40 per cent Montmorillonite and 20 per cent Illite. The clay fractions of C horizon was estimated to contain 50 per cent Montmorillonite and 20 per cent Illite.

The clay fractions of the Kirkland-1 and Kirkland-2 B horizons were estimated to contain 35 and 50 per cent Montmorillonite and 25 and 20 per cent Illite, respectively. The clay fractions of C horizons of the two soils were estimated to contain 40 per cent Montmorillonite and 30 and 35 per cent Illite, respectively.

The fractionation of the clay also afforded a study of the particle size ranges that were separated. Montmorillonite was found to be

the predominant clay mineral in the <0.1 micron fractions. Illite was usually the predominant clay mineral in the 2.0-0.6 and 0.6-0.1 micron fractions.

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